

Magnetic Behavior of Fe-Ni Compacts under Sintering Process

著者	KOJIMA Hiroshi
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Magnetic Behavior of Fe-Ni Compacts under Sintering Process

Hiroshi KOJIMA

The Research Institute for Scientific Measurements

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Synopsis

Sintering process of Fe-Ni compacts was observed by measuring their densities and magnetic properties. The troidal compacts of Fe-Ni alloy containing 45% to 85% of Ni were prepared from the hydrogen reduced Fe and Ni powders, which were obtained by the oxidation of their respective carbonyl compounds. Increase in density with rise in sintering temperature between 600°C to 1100°C was independent of their composition, but the magnetic saturation Bs of higher nickel compact approached the solid alloy's value more rapidly than lower nickel specimens.

I. Introduction

The sintering process of monometallic powdered compacts may be represented mainly by the elimination of voids and it can be observed by changes in density, electric conductivity and mechanical properties, etc. For bimetallic compacts, however, homogenization must also be noted, except in the case of sintering from fully alloyed powders. Of course, many reports have been made on the homogenization rate in sintering process. For instance, Rhines and his co-workers studied the sintering process of Cu-Ni compacts by measuring their mechanical properties and electric conductivity.⁽¹⁾⁽²⁾ The results of microscopic and X-ray observations in the same system were also given by Hijikata⁽³⁾.

In Fe-Ni system, for example, Hamprecht and Schlecht⁽⁴⁾ carried out a study of the carbonyl method and Hijikata⁽⁵⁾ made observations of thermal expansion.

In the alloy system forming a continuous series of solid solution, as in the above examples, the sintering process can be traced out easily, because the homogenization of these system is very simple, depending on the diffusion velocity between the components, as was discussed by Duwez and Jordan.⁽⁶⁾

From these points of view and also for the reason that their pure and fine powders are easily made by our method⁽⁷⁾, the relation between magnetic properties and sintering degree was studied in the γ -phase region of Fe-Ni system in the experiment. It is interesting to observe magnetic behaviors at every stage of

(1) F.N. Rhines, R. A. Colton in J. Wulff *Power Metallurgy* Cleveland (1942), 67.

(2) F.N. Rhines, R. A. Meussner in C.G. Goetzel *Treaties on Powder Metallurgy* Vol. 1 New York (1949), 553.

(3) K. Hijikata, J. Japan Inst. Metals **18** (1954), 244.

(4) G. Hamprecht and L. Schlecht, *Metallwirt.*, **12** (1933), 281.

(5) K. Hijikata, J. Japan Inst. Metals, **18** (1954), 247.

(6) P. Duwez and C. B. Jordan in W. E. Kingston *The physics of powder metallurgy* New York (1951), 230.

(7) T. Okamura, H. Kojima and Y. Kamata, J. App. Phys., Japan, **21** (1952), 9.

sintering, but there are few reports on it, as pointed out by Ogawa and his co-workers in their report on the sintering velocity of Cu-Ni compacts.⁽⁸⁾

We obtained interesting results on the magnetic properties of sintered iron, which was prepared from $\text{Fe}(\text{CO})_5$ ⁽⁹⁾. Therefore, the same procedure was adopted in Fe-Ni system. The γ -phase region of Fe-Ni system, as a high permeable material, has so varied uses that it is also of some significance industrially to study their magnetic behavior during sintering.

II. Preparation of specimens

$\text{Fe}(\text{CO})_5$ and $\text{Ni}(\text{CO})_4$ were obtained from hydrogen-reduced iron and nickel under the high pressure of carbon monoxide gas and they were purified by distillation for several times. Then the compounds were oxidized with the oxidizing apparatus, reported before by the author.⁽¹⁰⁾

Iron oxide powder thus obtained was dark red and nickel oxide was black and mean diameter of both oxides was about 0.1μ . These oxides were reduced to metallic powder by heating them at 450°C for 5 hours in dry hydrogen stream. After weighing five per cent of each powder from 45% to 85% Ni, both powders were mixed in an automatic mortar. Troidal specimens were pressed from these mixed powders under the pressure of 1 ton/cm^2 . Outer and inner diameters of the troid were 32 mm and 20 mm respectively, and about 5 mm high and about 11 g in weight.

III. Experimental procedure

Nine specimens thus prepared were heated at 600° , 800° , 1000° and 1100°C for an hour in dry hydrogen stream. Then, the furnace was removed in order to cool the troids rapidly.

After taking the measurements of their densities and magnetic properties, specimens were heated for another hour, and the procedure was repeated till the fifth measurement. But the specimens, heated above 800°C , were pre-sintered at 600°C for 5 hours before they were subjected to the above procedure. The density of the specimens was calculated from their volume and weight, and the magnetic properties were measured by the ballistic galvanometer method.

IV. Results obtained

In Fig. 1, the changes in density are plotted against nickel content for various sintering conditions.

The changes are linear to the composition and the lines represent from the bottom, the density of troid, heated at 600°C for 1 hour, at 600°C for 5 hours, at 800°C for 1 hour and at 1100°C for 5 hours respectively. The uppermost solid line is the density for each composition without any pore, calculated from the data

(8) S. Ogawa, J. Mizuno T. Hirone and S. Ogi, Sci. Rep. RITU, A2 (1950), 780.

(9) T. Okamura, H. Kojima and Y. Kamata, Sci. Rep. RITU, A3 (1951), 748.

(10) H. Kojima, Rep. Res. Inst. Sci. Meas., Tohoku Univ. 2 (1952), 101.

obtained by X-ray observation.

The density of the specimens, heated at 1100°C for 5 hours is about 98% of the density of block metal, but, when heated at 600°C it is only 55% for an hour. The ratio of the apparent and true density is independent of the composition, and all the lines in Fig. 1 are almost parallel.

The relations of density and sintering time at various sintering temperatures are shown in Figs. 2 and 3 for 45% and 85% Ni respectively. The uppermost solid lines in these figures are also the density of block metal at each composition. The density of compact approaches a certain value with an increase in sintering time to which will be determined by composition and sintering temperature.

In Fig. 4, the relation between sintering temperature and density is re-plotted from the above results for 45% Ni, for an example. The density of compacts increases rapidly to the density of block metal with a rise in sintering temperature.

The ratio of apparent and true density has become 98% after the sintering at 1100°C for 5 hours and the diminishing velocity of voids is much greater than the sintering rate by any other methods.

For example, Rostker⁽¹¹⁾ obtained the Fe-Ni compact of about the same density after sintering at

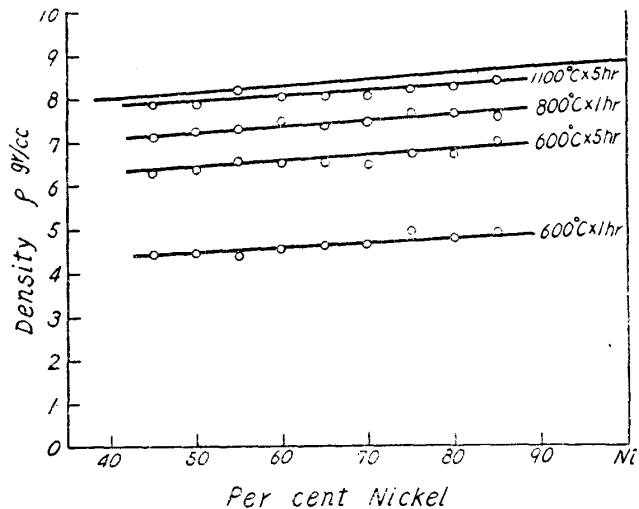


Fig. 1. Density as a function of nickel content in Fe-Ni compacts sintered under various conditions.

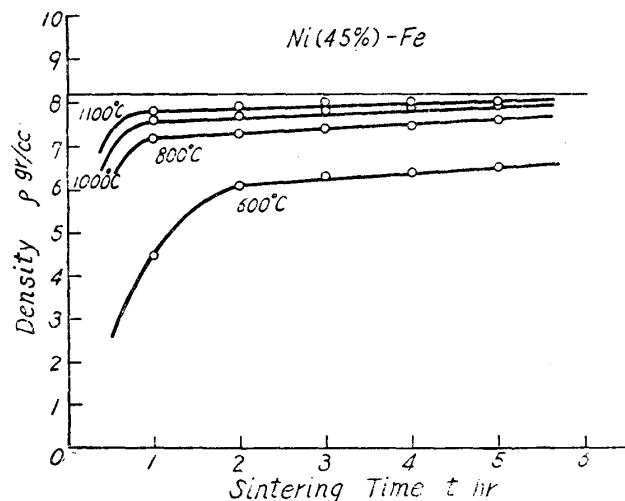


Fig. 2. Density of 45% Ni compact as a function of sintering time.

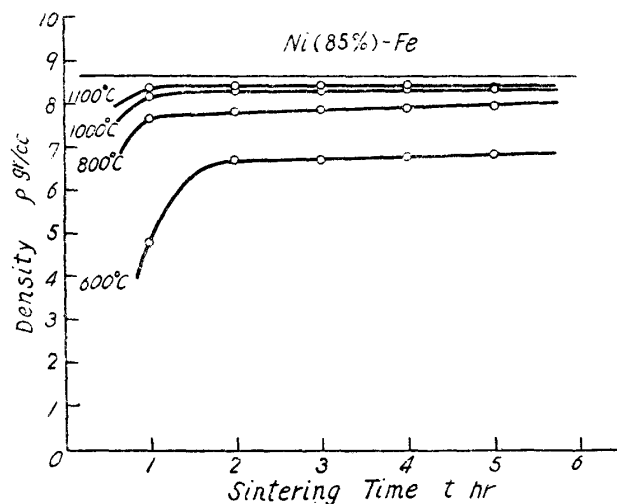


Fig. 3. Density of 85% Ni compact as a function of sintering time.

(11) W. Rostker, Trans. Am. Inst. Mining Met. Engrs., 180 (1949), 672.

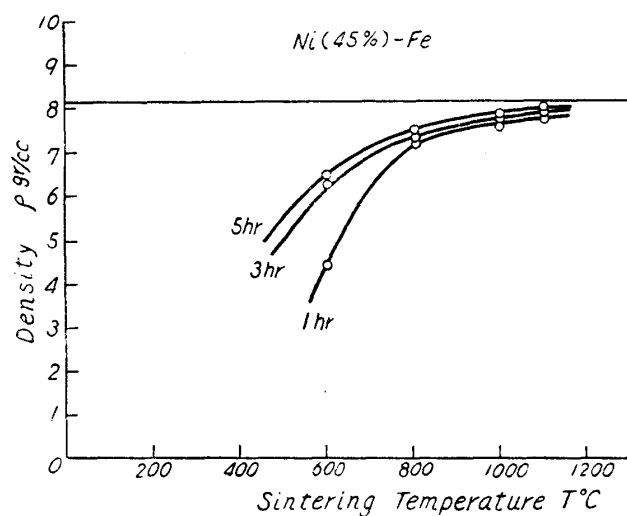


Fig. 4. Density of 45% Ni compact as a function of sintering temperature.

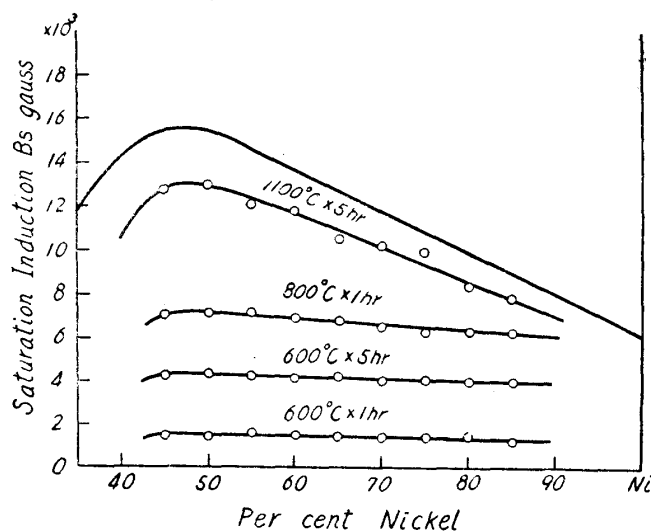


Fig. 5. Saturation induction as a function of nickel content in Fe-Ni compacts sintered under various conditions.

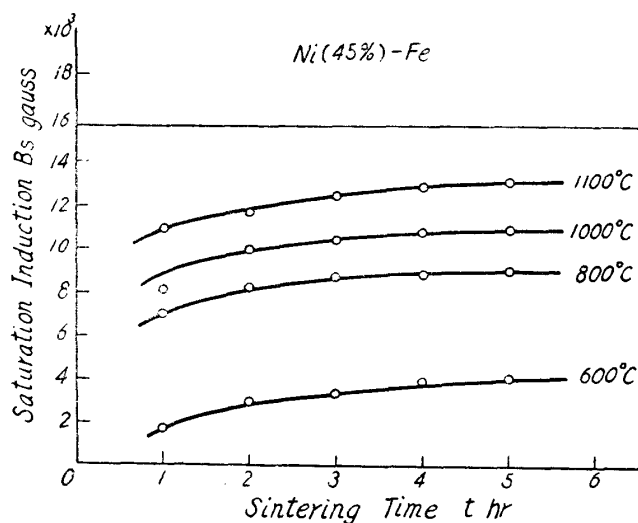


Fig. 6. Saturation induction of 45% Ni compact as a function of sintering time.

1400°C for 24 hours, which was made of carbonyl nickel and electrolytic iron powder.

This fact shows great sintering capacity of used powders due to their small particle size and great surface activity.

Fig. 5 illustrates the relation between saturation induction B_s and the composition under various sintering conditions. The uppermost solid line shows B_s for block metal as in the above figures⁽¹²⁾.

It is seen in this figure that the increase in B_s of the compacts, heated at 800°C for 1 hour is almost independent of their composition, and the curve becomes nearly parallel to the uppermost solid line in sintering at 1100°C for 5 hours. The ratio of apparent and true saturations, B_s'/B_s , is about 80% in lower nickel and 90% in higher nickel specimens. It may be due to the larger homogenizing velocity of the specimen, mixed with smaller amount of alloying element in diffusion.

The relations between B_s and sintering time for 45% and 85% Ni are illustrated in Fig. 6 and 7 respectively. The difference in homogenization rates due to composition is also seen clearly in these figures.

The changes in maximum permeability μ_m are plotted against composition in Fig. 8. The solid and dotted lines show the μ_m of block metal in annealed and quenched state, and the scale of

(12) R.M. Bozorth, *Ferromagnetism*, New York (1951), 109.

both lines is plotted in the right side. In the figure, it is seen that the ratio of apparent and true maximum permeability μ'_m/μ_m is only about 10% in the compacts, sintered at 800°C for 1 hour, and the composition dependency of μ_m is remarkable in the specimens, heated at above 1000°C.

The relation between μ_m and sintering time are illustrated in Figs. 9 and 10 in 45% and 85% nickel respectively. Different from the changes in density or in saturation, μ_m of the compacts gradually increases with sintering time.

Fig. 11 shows the relation between the coercive force H_c and the composition. The solid and dotted lines in the figure show the coercive force of block metal in annealed and quenched state in scale on the right side. Like permeability, H_c'/H_c comes to about 10~2 in this figure, but the difference of H_c due to composition is less remarkable than μ_m . The decreases in coercive force with sintering time are shown in Figs. 12 and 13 in 45% and 85% nickel specimens. It is found that the coercive force of troid sintered at 600°C is very great, owing to large porosity.

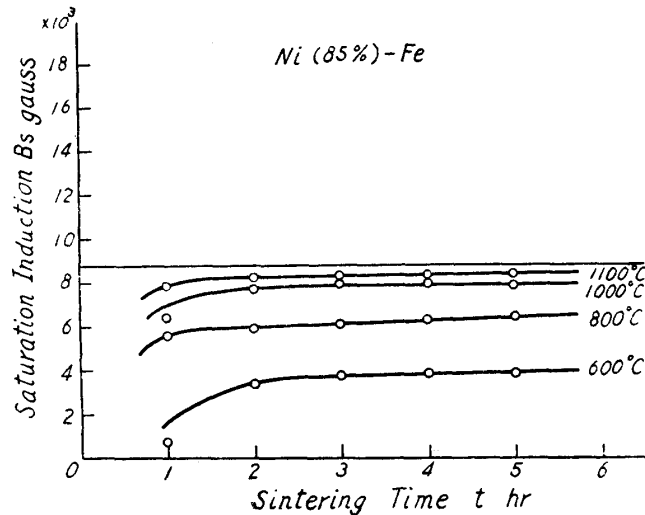


Fig. 7. Saturation induction of 85% Ni compact as a function of sintering time.

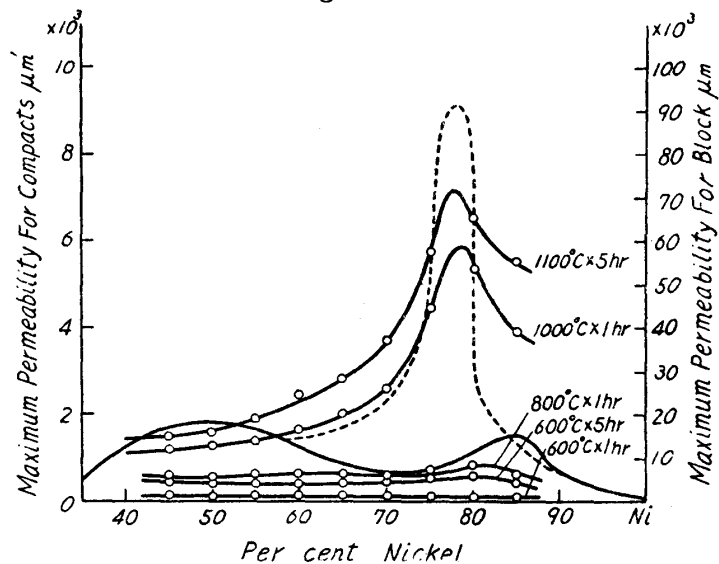


Fig. 8. Maximum permeability as a function of nickel content in Fe-Ni compacts sintered under various conditions.

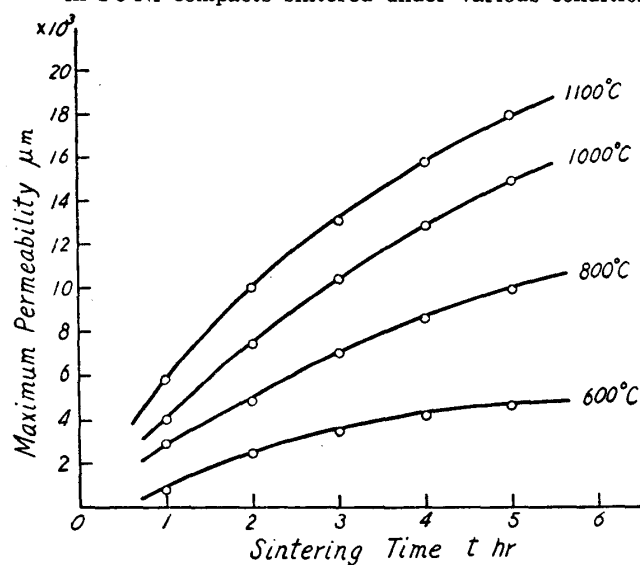


Fig. 9. Maximum permeability as a function of sintering time in 45% Ni compact.

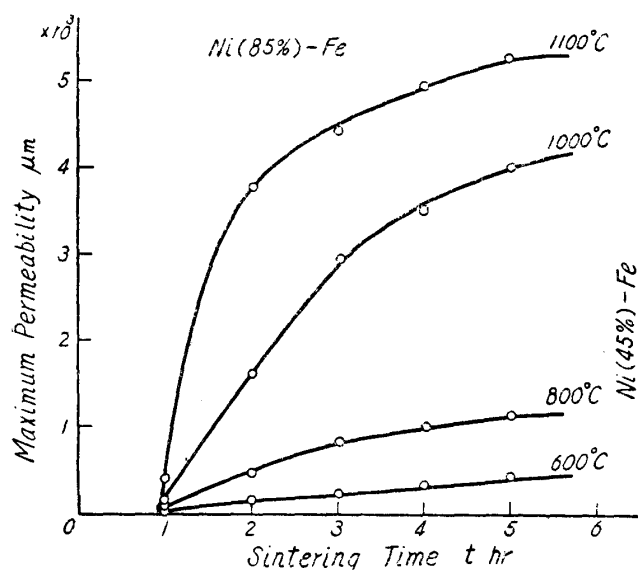


Fig. 10. Maximum permeability as a function of sintering time in 85% Ni compact.

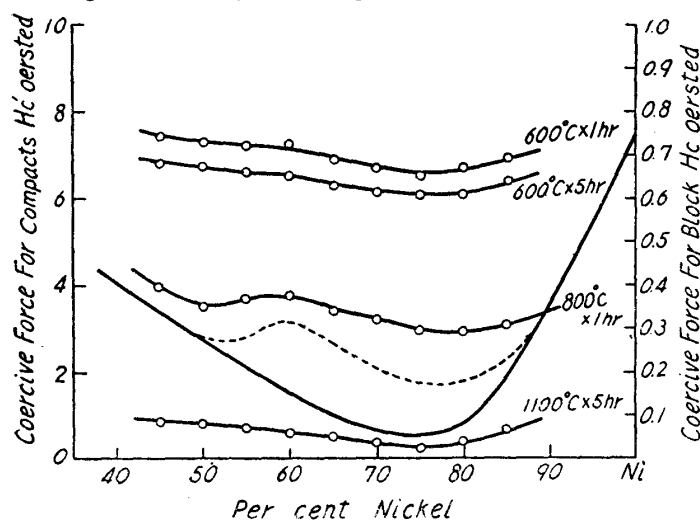


Fig. 11. Coercive force as a function of nickel content in Fe-Ni compacts sintered under various conditions.

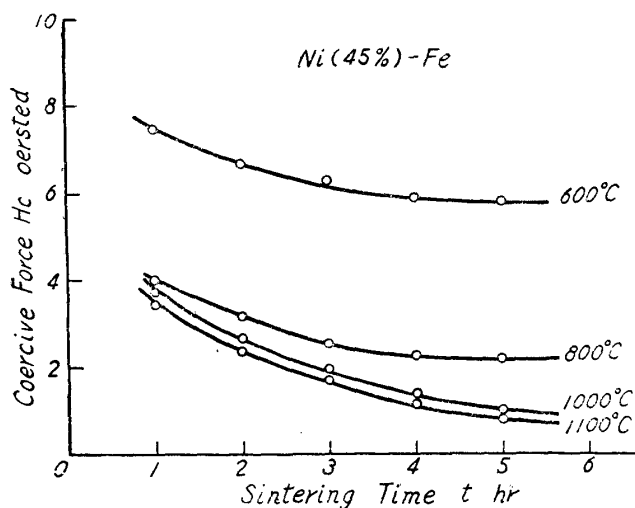


Fig. 12. Coercive force as a function of sintering time in 45% Ni compact.

V. Discussion

Different from the sintering of iron powder,⁽⁹⁾ the increase in density with sintering temperature is monotonous and abrupt. The difference in increasing rate of the density of Fe-Ni compacts could not be found in the experiment, as mentioned above. That is to say, the eliminating rate of voids is almost independent of the composition of the compacts to be sintered in mixture of two metallic powders of equal size. On the other hand, the rate of apparent and true induction saturations, B_s'/B_s , is smaller in the compacts with smaller iron content. In other words, it may be recognized from the observation of saturation induction that the larger nickel content, the greater the homogenizing rate.

The changes in Curie points during the sintering were studied in Ni-Cu compacts by Ogawa and his co-workers, but no remarkable results were reported⁽⁸⁾. The saturation value B_s of Fe-Ni compacts has a fairly good correspondence to the sintering stage in this experiment. In order to make it clear, the values of B_s'/B_s are plotted against the porosity of compacts in Fig. 14.

It can be seen in the figure that the troid of 85% Ni has a higher value of B_s'/B_s than the one of 45% Ni in the same porosity.

The relations between porosity and μ'_m/μ_m or H_c'/H_c for the 85% and 45% Ni compacts are shown in Figs. 15 and 16 respectively.

Differing from the case of B_s , the changes in coercive force H_c and maximum permeability μ_m have no obvious correspondency to the homogenization velocity or the elimination rate of voids. And it is reasonable, because they are apt to be affected greatly by grain size, etc.

The porosity in the specimen have so great effect upon the value of μ_m and H_c that the ratio is only 20% or so even for the compacts of about 2% porosity.

The abnormally high value of μ_m for the compacts of 75%~80% Ni in Fig. 8 may be due to the rapid velocity of cooling in the sintering process, and it is not the composition dependency of the sintering velocity but essential properties of permalloy.

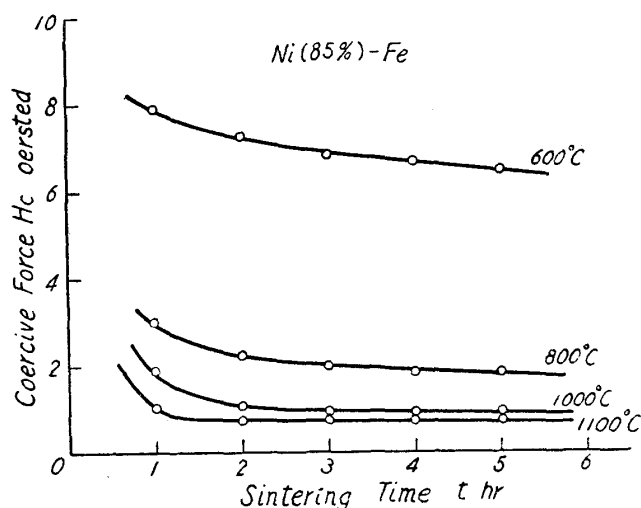


Fig. 13. Coercive force as a function of sintering time in 85% Ni compact.

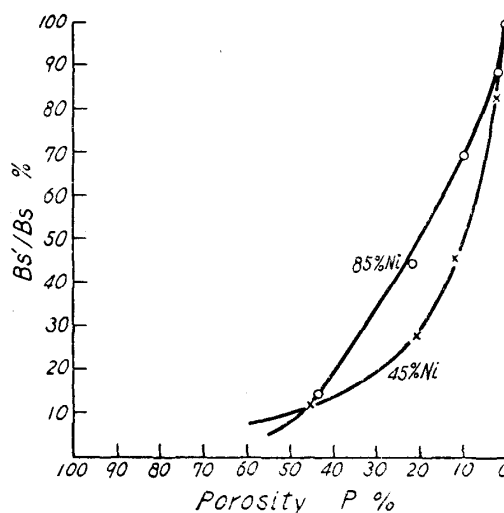


Fig. 14. The ratio of the saturation induction of the 45% and 85% Ni compacts to that of the massive substances as functions of porosity.

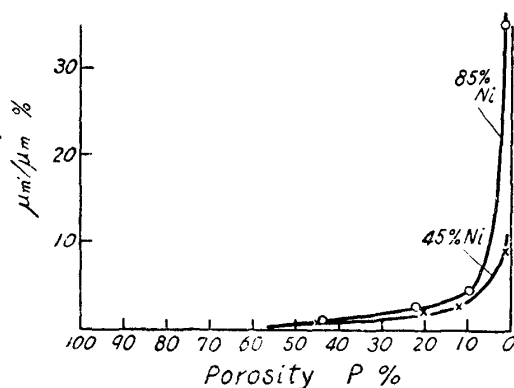


Fig. 15. The ratio of the maximum permeability of the 45% and 85% Ni compacts to that of the massive substances as functions of porosity.

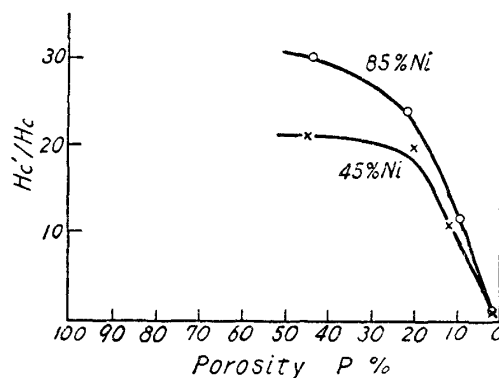


Fig. 16. The ratio of the coercive force of the 45% and 85% Ni compacts to that of the massive substances as functions of porosity.

Summary

Fe-Ni compacts of 45%~85% Ni were prepared from iron and nickel oxide powder, obtained by the oxidation of $\text{Fe}(\text{CO})_5$ and $\text{Ni}(\text{CO})_4$ respectively. Then they were heated at 600~1100°C for 1~5 hours in dry hydrogen stream and the measurements of density and magnetic properties were made at each sintering stage. The following results were obtained from the observation:

(1) The increases in density in these compacts are independent of their composition, and the rise is abrupt but monotonous against the sintering temperature.

(2) After the sintering at 1100°C for 5 hours, the apparent density of compacts amounts to 98%.

(3) The increasing velocity of B_s against the sintering temperature is larger in the compacts of higher nickel content and the ratio of B_s'/B_s amounts to about 90% after the sintering at 1100°C for 5 hours.

(4) From the above results, it can be seen that the homogenization rate is larger in the higher nickel compacts.

(5) The value of μ'_m/μ_m amounts to only 20% or so even in the compacts of about 98% density.

(6) The rise of μ_m by quenching effect is recognized in the compacts of 75%~80% Ni, sintered above 1000°C.

(7) The value of H_c'/H_c amounts to 2.0 in the compacts of 98% density.

(8) The change in coercive force is almost independent of their composition.

After the sintering at 1100°C for 5 hours, the compacts can be forged or rolled easily. For example, after the forging about 50% at 800°C, they were cold rolled to 99% in thickness without annealing. It might be due to the effect of remaining oxide at grain boundary that the value of H_c and μ_m of the compacts were smaller in spite of their higher density.

In conclusion, the author wishes to express his hearty thanks to the late Prof. T. Okamura for his kind guidance, and also to Mr. C. Miyakawa for his assistances in the course of this work.